Interfacial Stability of Thin Film Hydrogen Sensors

2004 DOE Hydrogen, Fuel Cells & Infrastructure Technologies Program Review

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Objectives

Assist DOE in the development of hydrogen safety sensors

- Make available the technology to produce safe, reliable,sensitive, fast, lightweight, and inexpensive hydrogen safety sensors.
- Determine the factors limiting the lifetime and performance of thin film hydrogen sensors in realistic environmental conditions.
- Find ways of extending lifetime and functionality of thin film hydrogen sensors.

Budget

- Total project funding: $ 560 k
- Total project labor: ~ 2 FTE-years
- Funding in FY04: $ 80 k (~ 0.3 FTE-y)

Reviewers Comments from FY03

–***How much power for system?*** Detailed design delayed due to funding limitations.

–***Acknowledge difficulties.*** Interfacial stability of chemochromic layer and dissociation catalyst, response time at low temperature and high humidity.

–***Broaden materials investigation.*** Have developed alternate chemochromic materials and have researched Pd alloys for the dissociation catalyst.

–***Need fundamental work.*** Fundamental work on the protective layer was planned for FY04, but could not proceed due to funding limitations.

–***Keep up the good work.*** The work plan for FY04 would have made significant strides toward meeting DOE goals, commercializing the optical sensors, and addressing all of the review comments from FY03. The work has been delayed by funding issues during FY04.

Technical Barriers

Hydrogen Safety Barriers

- C. Validation of Historical Data
- D. Technical and Scientific Understanding of Systems Limits the Value of Protocols
- F. Liability Issues
- L. Expense of Data Collection and Maintenance
- M. Quality of Data

Fuel Cell Barriers

- B. Sensors for measurement and safety

Technology Validation Barriers

- C. Hydrogen Refueling Infrastructure (safety aspects)

Hydrogen Production Barriers

- E. Control and Safety

Hydrogen Delivery Barriers

- D. High Capital Cost of Pipelines

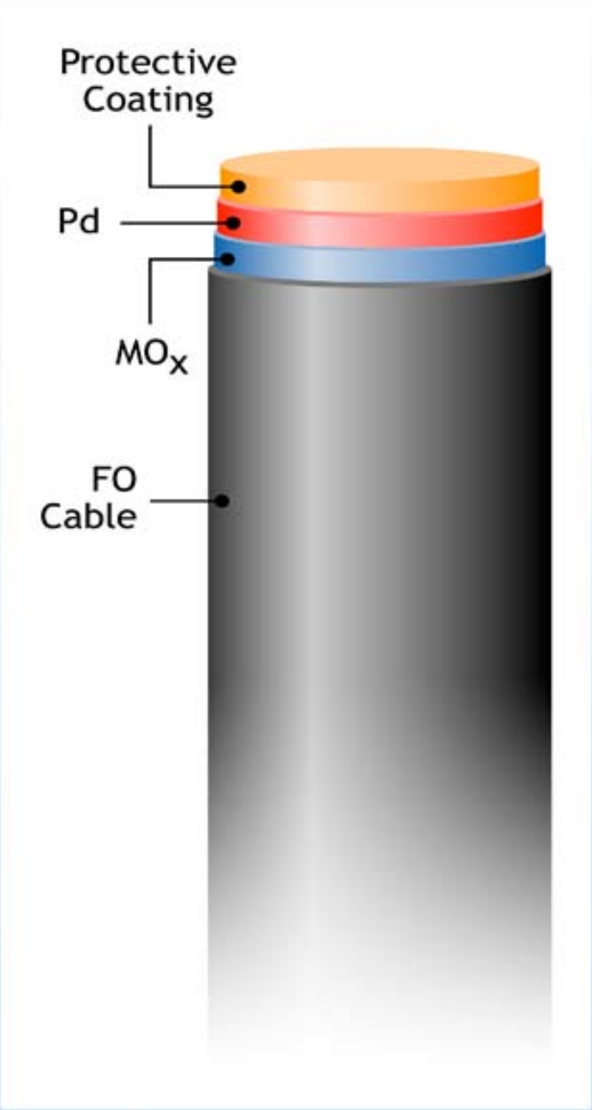
Technical Targets

(Safety Sensors)

- Measurement range: 0.1 – 10% H_2 in air
- Operating temperature: -30 – 80°C
- Response time: <1 s
- Accuracy: 5%
- Gas environment: ambient air, 10 – 98% RH
- Lifetime: 5 y
- Selectivity from interference gases, such as hydrocarbons, is needed.

Approach

- Investigate the stability of thin films that undergo optical changes in the presence of hydrogen.
- Construct test articles of thin films and subject them to environmental stresses.
- Measure response to environmental stresses and aging.
- Develop improved protective strategies.

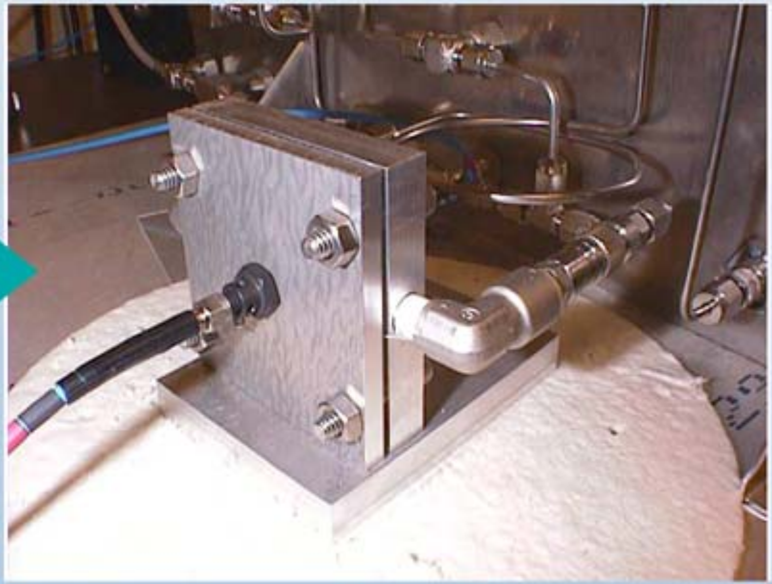


Fiber-Optic Sensor Testing

(Diode array spectrophotometer to measure T or R)

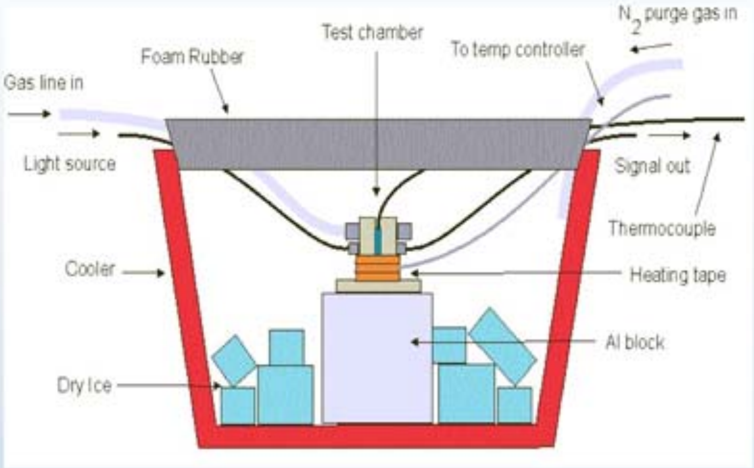
Gas manifold and test station

Test Chamber



Approach for Testing T and RH

- **Environmental chamber for T and RH studies.** Use dry ice plus proportional heaters to control T.
- **Gas manifold with MFCs for accurate gas proportioning.** Use saturated air proportioned to specific RH.
- **Conduct studies of sensor response with variation of T and RH.**



Project Safety

Conduct all experimental work in a safe manner

- Maintain Safe Operating Procedures for all laboratory activities, update ever year. Conform to all DOE and OSHA standards for laboratory practice.
- Use engineering controls to provide safety in the handling of hydrogen, including the use of non explosive gas mixtures for testing, the use of dilute (below the lower exposure limits) contaminant gas mixtures in, and the use of ventilated enclosures for storage and testing.
- Satisfy Laboratory Safety Analysis Reviews and conduct quarterly lab safety inspections.

Timeline

Feasibility study of WO_3/Pd thin film sensor configuration.

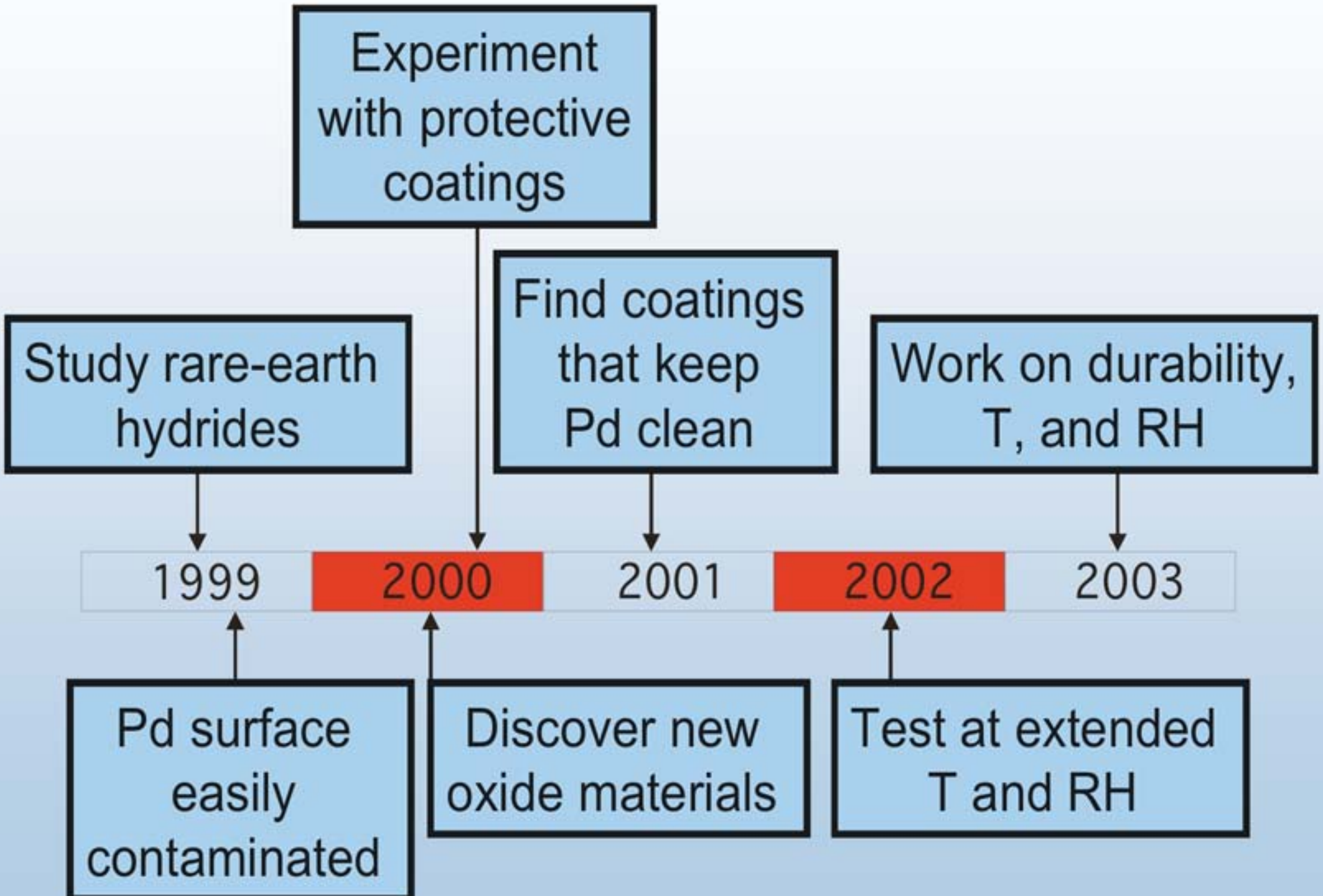
Surface Plasmon configuration is faster and more sensitive, but requires additional elements and is prone to interference.

Use rare earth hydride materials (e.g., GdH_x, YH_x, $GdMg_xH_y$).

1994	1995	1996	1997	1998

Initial concept of Pd/WO_3/FO cable end.

Timeline, cont'd.

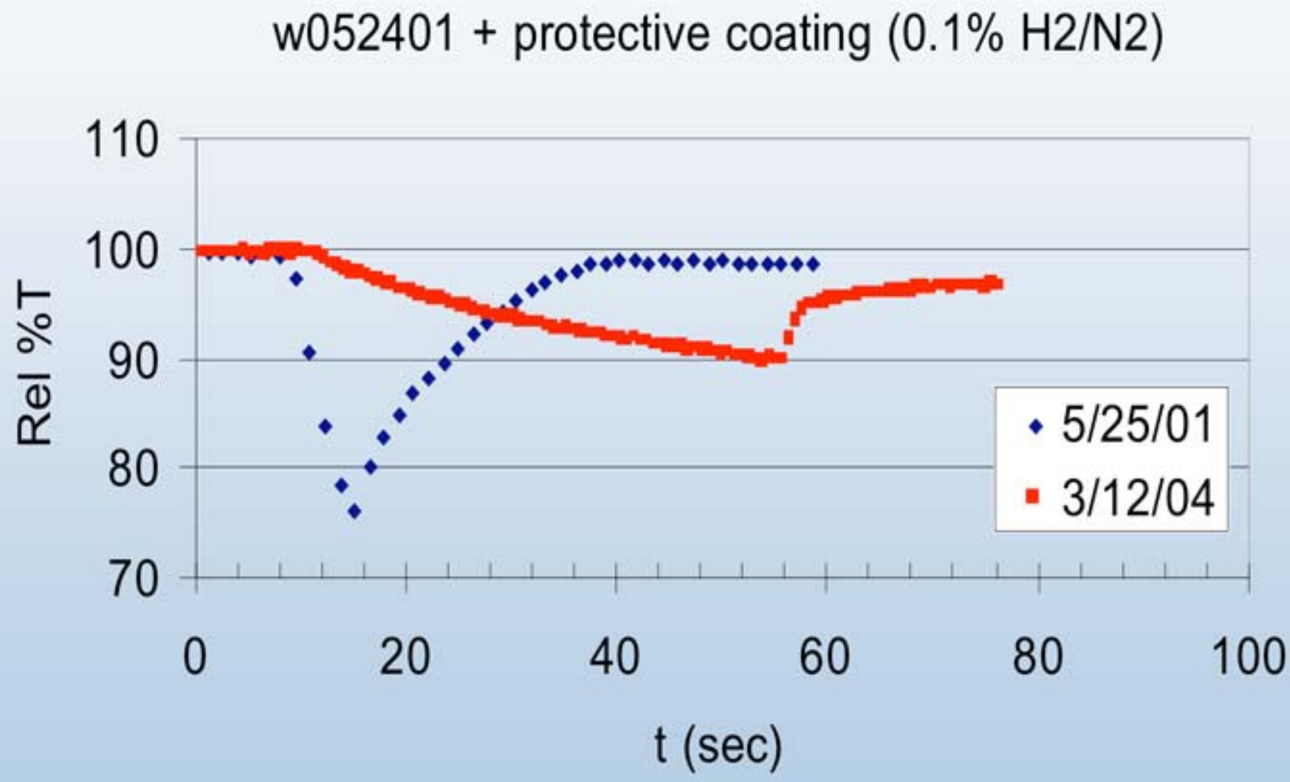


Technical Accomplishments/Progress

- Tested optical sensor configurations that meet most DOE criteria for safety sensors.
- Extended sensor lifetimes by an additional year (now have sensors that have been operable for a total of 3 years).
- Analyzed subtle compositional differences in protective coatings that result in dramatic changes in performance.
- Provided support for the Bio-Hydrogen project by constructing large area sensor plates for semi-quantitative evaluation of hydrogen producing algae.

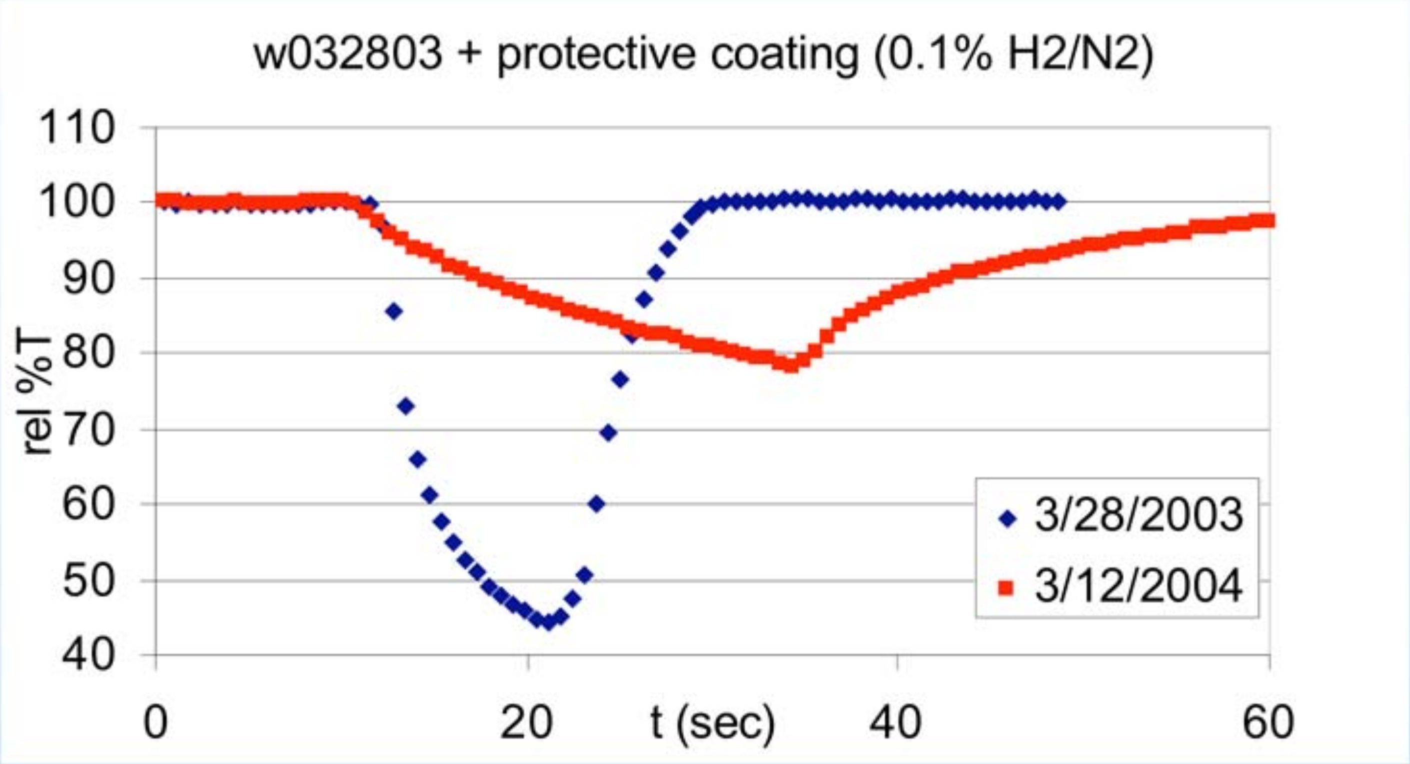
Sensor Functions after 3 Years

(operating at the detection limit of 0.1% H_2)



Sensor Function after 1 year

(at the detection limit, 0.1% H_2)



Response time < 1 sec at the detection limit after one year.

Interactions and Collaborations

GVD Corporation

- Advanced polymeric coatings for biohydrogen sensor plates

Element One

- Materials for wide-area visual hydrogen sensors

Davidson Instruments

- Prototype fiber optic sensor design

PNNL

- Fundamental research of materials to improve performance of protective coatings

All work suspended due to lack of funding

Future Work

- Investigate the fundamental behavior of the protective coatings in order to optimize performance.
- Analyze the response of optical sensors coated with protective polymeric CVD films when exposed to low temperature and high humidity environments.
- Design a control package for probing the status of the thin film optical sensors.
- Fabricate a prototype sensor and control package for testing.